DEVELOPMENT OF A NOVEL LASER MATERIAL FOR MINIATURIZED LASER SYSTEMS

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Crystals of NdP_5O_{14} (NPP), $NdLiP_4O_{12}$ (LNP), and $Nd_xY_{1-x}Al_3(BO_3)_4$ (NAB) have been grown from high-temperature fluxed solutions. LNP crystals of good optical quality could only be grown on the surface of the flux, indicating that top seeding methods might be the most suitable for obtaining large crystals for fabrication of laser rods. Crystals of NAB were small and contained flux inclusions. Other fluxes and various Nd:Y ratios will be used in future experiments to determine if optically clear single-phase

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20. ABSTRACT (Cont'd.)

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Fluorescence lifetime and transmission spectra of NPP, LNP, and NAB crystals grown in-house have been measured. The results of these measurements compare favorably with the reported values in the literature. Except in the case of NAB, we have observed increased background absorption in the shorter wavelength region of the visible spectrum. It is believed that this behavior of NAB may be caused by the presence of Pb in the crystal. The experimental laser setup designed previously has been completed. During initial experiments, we measured the output energy per pulse, and observed the transverse-mode structure of the beam and temporal behavior of the output pulse.

PREFACE

This work is being performed by Philips Laboratories, a Division of North American Philips Corporation, Briarcliff Manor, New York under the overall supervision of Dr. Rameshwar Bhargava, Director, Exploratory Research Group. Dr. Walter Zwicker, Senior Program Leader for Crystal Growth and Materials Technology, is the Principal Investigator. Mr. Emil Abelaf and Mr. Theodore Kovats are responsible for crystal growing; Dr. Sel Colak and Mr. Jacob Khurgin are responsible for materials evaluation as well as laser design and construction.

This program is sponsored by the Defense Advanced Research Projects Agency (DARPA) and was initiated under Contract No. MDA903-81-C-0034. Dr. Jefferey L. Paul is the Contracting Officer's Technical Representative for DARPA.

The work described in this second Quarterly Technical Report covers the period from 1 January to 31 March 1981.

SUMMARY

Crystals of NdP_5O_{14} (NPP), $NdLiP_4O_{12}$ (LNP), and $Nd_xY_{1-x}Al_3(BO_3)_4$ (NAB) have been grown from high-temperature fluxed solutions. LNP crystals of good optical quality could only be grown on the surface of the flux, indicating that top seeding methods might be the most suitable for obtaining large crystals for fabrication of laser rods. Crystals of NAB were small and contained flux inclusions. Other fluxes and various Nd:Y ratios will be used in future experiments to determine if optically clear single-phase crystals can be grown which are large enough for fabrication of laser rods.

Fluorescence lifetime and transmission spectra of NPP, LNP, and NAB crystals grown in-house have been measured. The results of these measurements compare favorably with the reported values in the literature. Except in the case of NAB, we have observed increased background absorption in the shorter wavelength region of the visible spectrum. It is believed that this behavior of NAB may be caused by the presence of Pb in the crystal. The experimental laser setup designed previously has been completed. During initial experiments, we measured the output energy per pulse, and observed the transverse-mode structure of the beam and temporal behavior of the output pulse.

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1. INTRODUCTION

Based on a survey covering optical properties and crystal growth of all better-known stoichiometric neodymium compounds (see Qtly. Tech. Report Nov-Dec 1980), it was decided to concentrate our efforts on three materials, viz., NdP_5O_{14} (NPP), $LiNdP_4O_{12}$ (LNP), and $NdAl_3(BO_3)_4$ [NAB].

The equipment setup for growing single crystals of these compounds was completed, and crystals of all three compounds were produced. Only crystals of NPP were of good optical quality and large enough for fabrication of laser rods. The only existing difficulty is that crucibles of proper shape, essential for growth of NPP crystals, are difficult to obtain. LNP crystals of good optical quality, but too small for fabrication of laser rods, were grown in the form of platlets on top of the melt. Top seeding experiments were therefore started. Crystals of NAP were of poor optical quality and mostly small. It is hoped that the addition of yttrium, replacing neodymium in the lattice, may improve the quality of these crystals.

The results of the optical evaluation of laser crystals and the output behavior of a NdP_5O_{14} miniature laser are given in Section 3. The absorption bands and fluorescence lifetime data for Nd^{3+} in three different crystals are given in Section 3.1. Section 3.2 describes the mechanical setup of the miniature laser system and the flash-lamp circuitry for discharging the lamp. Section 3.3 summarizes the initial results obtained for the output of this laser using a NdP_5O_{14} crystal. In each section, discussions are presented on the observed behavior of the crystals and the laser.

2. MATERIAL PREPARATION AND CRYSTAL GROWTH

2.1 NdP_5O_{14} (NPP)

Crystals of NPP (see Fig. 1) were grown on a routine basis using a previously described technique [Ref. 1]. Laser rods (2 x 2 x 20 mm) can be produced from such crystals. Equipment for this growth is inexpensive, and minimum supervision is needed during the growth. It is believed that such NPP miniature laser rods will be superior in performance to Nd:YAG in small hand-held laser devices and could be produced appreciably cheaper than Nd:YAG rods delivering similar output powers.

A difficulty has developed recently during the crystal growth. The bottoms (normally flat) of the vitreous carbon crucibles obtained in the last shipments had a slightly convex shape on the inside; this caused the crystal seed to drift during growth, from the center toward the wall of the crucible. This hinders proper growth of the crystal, causing irregular growth and the development of strains (see Fig. 2). This deformation of the crucibles was caused by changes in the manufacturing technique of the only supplier, a French company. Discussions were held with an American company to specially fabricate and supply us with crucibles of proper shape. In the meantime, inserts for the old crucibles are being fabricated to prevent this seed drift. Several laser rods (2 x 2 x 20 mm) fabricated from one of our NPP crystals are presently being optical characterized, and lasing experiments (Section 3) are being performed.

2.2 $NdLiP_4O_{12}$ (LNP)

Because LNP melts incongruently, the crystals have to be grown by a slow cooling method from a flux or by top-seeding techniques (Kyropoulos Technique) [Ref. 2]. Our starting materials, Li-carbonate, Nd-oxide and ammonium-dihydrogen phosphate, were fused in steps up to 1000°C in a platinum crucible. The molar ratios of the resulting oxides Li_2O:Nd_2O_3:P_2O_5 varied between 40-44:1-5:50-53. Attempts were made to grow crystals by slow cooling of these melts at rates of 0.5 - 2°C/hour. Slow cooling rates and low Nd_2O_3 concentrations produced the best results. Since the melt tends to supersaturate, the crystallization occurs spontaneously, at a certain point, resulting in poor-quality crystalline aggregates.

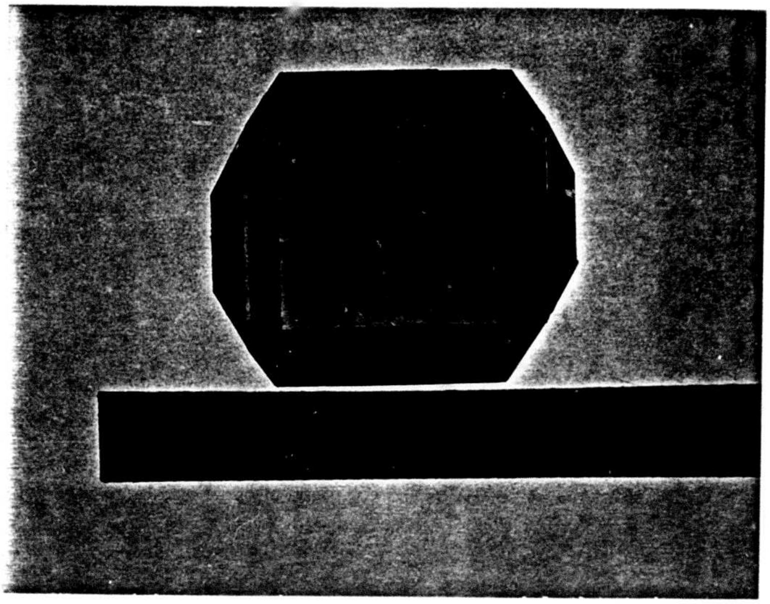


Figure 1. Crystal of NPP grown in center of crucible.

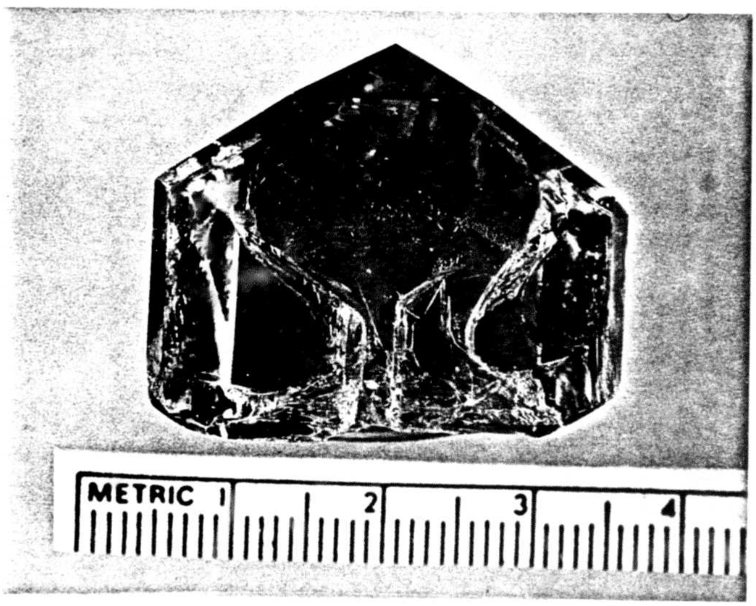


Figure 2. Crystal of NPP grown toward wall of crucible.

Although lower Nd_2O_3 concentrations seem to minimize this effect, crystals inside the flux were of poor quality (see Fig. 3).

The only crystals of good optical quality grew on top of the melt in the form of platelets up to about 0.5 x 5 x 10 mm (see Fig. 4). The platelets were used for optical evaluation studies (see Section 3). The results indicate that the system lends itself well for top-seeded growth. To do this, two vertical-tube furnaces were designed, built, and put into operation. The furnaces are wound with "super kanthal" and have an operating maximum temperature of 1200°C. To facilitate top-seeding, a "Crystar" puller was installed on one furnace; a second puller will be installed in the future. The puller was slightly modified to make it more suitable for operation; a sensor was added so that the stability of the pulling rate during crystal growth could be recorded.

The temperature control systems for the furnaces are presently being installed, and actual pulling experiments will be started when all of the system parts are received.

2.3 $NdAl_3(BO_3)_4$ (NAB)

NAB also melts incongruently at 1220°C, and crystals have to be grown, therefore, from high-temperature fluxed solutions. For this purpose, two vertical platinum tube furnaces were built and put into operation.

Platinum (with 5% gold) crucibles were ordered and received together with platinum liners for protecting the alumina furnace tubes. A suitable plantinum baffle system was made for insertion in the furnace tubes.

Two laboratory muffle furnaces were also made available for exploratory growth of $NdAl_3(BO_3)_4$. Table 1 summarizes the data for six fluxed-melt systems which have been used to grow rare-earth borates. When the rare-earth (RE) is Nd, the number of fluxes is reduced to four. It can be seen that only the BaO, B_2O_3 combination will lead to a system that is really suitable for top seeding.

Table 2 summarizes the exploratory experiments with NAB. The first experiment (melt #1) produced crystals of $NdAl_3(BO_3)_4$ up to 1 cm in size

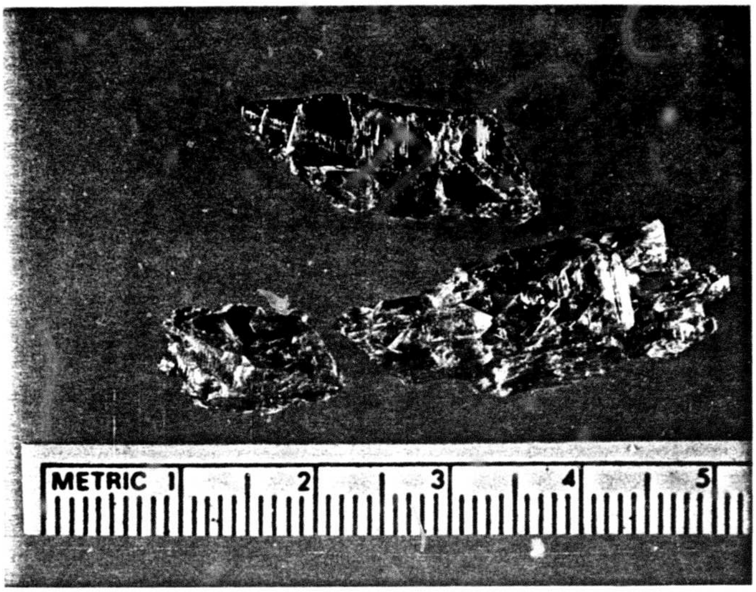


Figure 3. Crystals of LNP with flux inclusions.

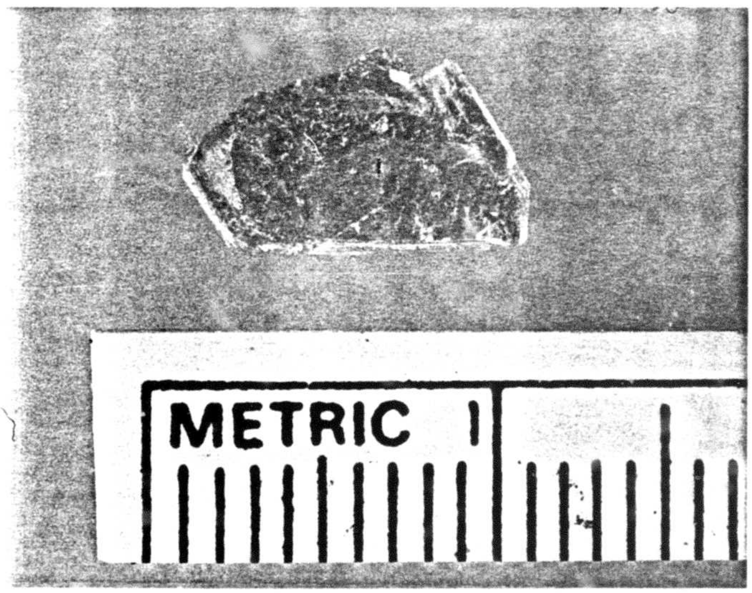


Figure 4. Single-crystal platelet of LNP grown on surface of flux.

TABLE 1: Fluxed Melts Used for Growth of Neodymium Aluminum Borates.

Flux	Nd
$Li_2O:B_2O_3$	V
$K_2O:3MoO_3$	V + I
$BaO::B_2O_3$	V + G
$PbO:B_2O_3$	X
$PbF_2:B_2O_3$	V
$Bi_2O_3:B_2O_3$	X

V crystal growth
X no crystal growth
I Mo incorporated in crystals
G melt suitable for top-seeding

TABLE 2: List of Exploratory Experiments with NAB.

Melt No.	Crystal	Flux
1	$NdAl_3(BO_3)_4$	$PbF_2:B_2O_3$
2	$NdAl_3(BO_3)_4$	$Bi_2O_3:B_2O_3$
3	$NdAl_3(BO_3)_4$	$BaO:BaF_2:B_2O_3$
4	$Nd_xY_{1-x}Al_3(BO_3)_4$	$PbO:B_2O_3$
5	$Nd_xY_{1-x}Al_3(BO_3)_4$	$PbO:B_2O_3$
6	$NdAl_3(BO_3)_4$	$PbO:B_2O_3$

(see Fig. 5). The crystals do not appear optically clear, however, which may be partially due to flux inclusions and partially due to the presence of two phases. The melt composition is given in Table 3. A 100 ml crucible was placed in the usual LPE furnace; crystal growth occurred at 1020°C. It was also possible to do some top seeding experiments, although the PbF_2 is really too volatile for this work. The real problem was the two-phase nature of the crystals as seen by X-ray diffraction. This is similar to that reported by Jarchow et al. [Ref. 3] who show that their crystals of $NdAl_3(BO_3)_4$ had C2/c and C2 phases. It maybe that their crystals contain Nd aluminum dimetaborate, a P6/mmm class material as reported by D. Yu. Pushchrovskii et al. [Ref. 4]. This phase is obtained when the supersaturation is too high. This two-phased nature of the crystals is being studied at the present time. The other problem is that this melt composition produces a second phase as well as the crystals. This phase, now identified as $9Al_2O_3$, $2B_2O_3$, is caused by too-high a B_2O_3 content in the melt or too high a $NdAl_3(BO_3)_4$ concentration. This may also be a clue to the two-phase nature of the crystals if this aluminum borate grows into the crystals. An experiment is running at the present time with less B_2O_3 in the melt.

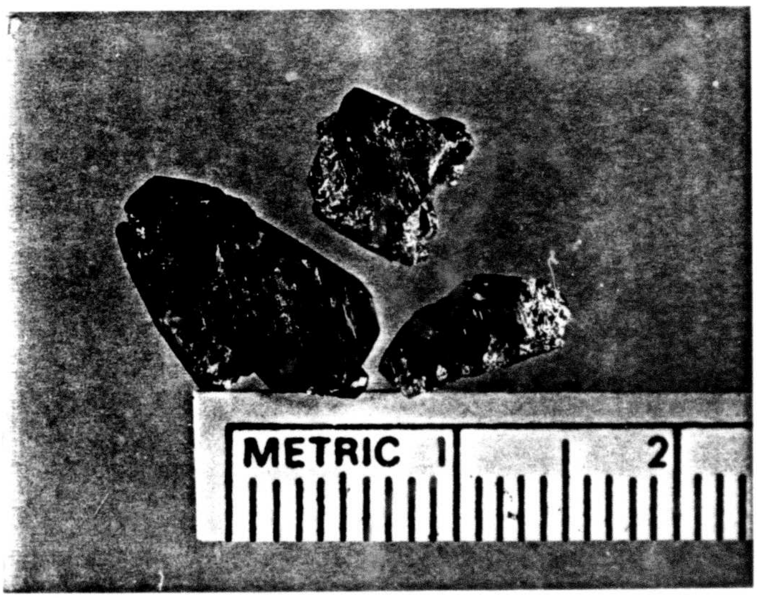


Figure 5. Crystals of NAB.

TABLE 3: Melt Compositions and Growth Temperatures in Exploratory Growth Experiments for NAB.

Melt #1	Nd_2O_3	36.52 g	
	Al_2O_3	33.21 g	Pure oxides.
	B_2O_3	60.20 g	100 ml crucible.
	PbF_2	120.05 g	T growth 1020°C.
Melt #2	Nd_2O_3	15.31 g	
	Al_2O_3	13.92 g	Premelted in O_2.
	Bi_2O_3	84.80 g	Ordinary purity oxides.
	B_2O_3	73.29 g	100 ml crucible.
Melt #3	Nd_2O_3	40.38 g	
	Al_2O_3	36.72 g	Premelted at 1160°C.
	BaO	36.82 g	Homogenization temperature 1200°C.
	BaF_2	18.76 g	Growth temperature 1090 -> 900°C.
	B_2O_3	66.84 g	
Melt #4	Nd_2O_3	8.21 g	
	Y_2O_3	11.02 g	Homogenization Temp. = 1100°C.
	Al_2O_3	21.82 g	Growth Temperature 1050 -> 930°C.
	PbO	76.32 g	at 1°C/hour.
	B_2O_3	95.52 g	3N, 4N and 5N purity chemicals.
Melt #5	Nd_2O_3	16.42 g	
	Y_2O_3	5.51 g	Premelted at 1150°C
	Al_2O_3	21.52 g	Growth 1040 -> 950°C
	PbO	76.32 g	at 1°C/hour.
	B_2O_3	95.52 g	3N, 4N and 5N purity chemicals.
Melt #6	Nd_2O_3	24.63 g	Premelted at 1150°C
	Al_2O_3	21.82 g	Growth 1040 -> 950°C
	PbO	76.32 g	at 1°C/hour.
	B_2O_3	95.52 g	3N, 4N and 5N purity chemicals.

The lifetime of these crystals grown from the PbF_2 flux was found to be 16 μs (see Sect. 3) which is in very good agreement with the literature [Ref. 5].

A melt using Bi_2O_3 (#2 Table 3) was used in an attempt to grow $NdAl_3(BO_3)_4$. If such a growth were successful, it would be interesting from two points of view. The simultaneous incorporation of the Bi^{3+} ion can act as a sensitizer as, for example, in the case of $TbAl_3(BO_3)_4$ [Ref. 6], and also would be the largest ion that could possibly be substituted for the RE. Until now, only RE gallium [Ref. 7] and RE iron borates [Refs. 8,9] have been grown from this flux system, and not the aluminum borates. Using melt composition #2, crystal growth was attempted between 1100 and 950°C. This was unsuccessful and may be due to the too-low $NdAl_3(BO_3)_4$ concentration in the melt or that it just does not work for aluminum borates. This will be tried again with a higher $NdAl_3(BO_3)_4$ concentration in the near future.

With the melt composition based on BaO BaF_2 B_2O_3 (#3), an attempt was made to grow $NdAl_3(BO_3)_4$ and to top seed the melt. This run did not produce any crystals. It was interesting to note that this composition was successful in producing Eu and $TbAl_3(BO_3)_4$ crystals when using the same molar ratios. To obtain quick results, it is probably better to use the molar ratios published by Chinn and Hong [Ref. 10] which do not contain any BaF_2. One result was very clear from this experiment, viz., there was little evaporation and virtually no attack of the alumina furnace tubes; thus, the expensive platinum liners may not be necessary.

The last three exploratory experiments were based on the PbO, B_2O_3 flux combination. The reasons for doing this were: a) the evaporation rate is not too high, thus top-seeding could be attempted, b) the flux produces large crystals of good quality. A remark made by a Russian conferee at the ICCG-6 conference in Moscow suggested that the Nd-Y system would be a better laser crystal then the pure Nd compound. Three melts were therefore made in which the Nd/Y mole ratio was 1/3, 2/3 and pure Nd. There was a parallel experiment reported by Leonyuk et al. [Ref. 11] using a $K_2O.3MoO_3$ flux. Their results show that up to 72 mole % substitution of yttrium by neodymium was good. At higher concentrations, molybdates were formed. It should be noted that two other publications by the same authors [Refs.

12,13], reported that they were able to produce the pure $NdAl_3(BO_3)_4$ from the $K_2O.3MoO_3$ flux.

Melt #4 produced crystals on the bottom of the crucible. No second phases were found, and the lifetime was measured to be 40-60 μs depending on which crystal was used. Melt #5 produced some crystals and a polycrystalline mass. The lifetime was measured to be 34 ± 3 μs. Finally, melt #6 did not produce any crystals, only flakes in the melt. These flakes have to be analysed and the composition of the melt changed. One point that is clear, the mixed crystals produce lifetimes which are better from a laser point of view.

3. EVALUATION OF MATERIALS FOR LASING

As discussed previously [Ref. 14], the most promising laser materials with Nd^{3+} are NPP, LNP, and NAB. Some samples of these crystals have been grown in-house for initial optical characterization. Information on the growth, crystalline behavior, and visual appearance of these samples are given in Section 2. This section gives the results and discussions on the optical behavior of these samples and the behavior of NPP under lasing conditions. The NAB crystal used in the following evaluations were grown by using PbF_2:B_2O_3 (Melt #1) given in Tables 2 and 3.

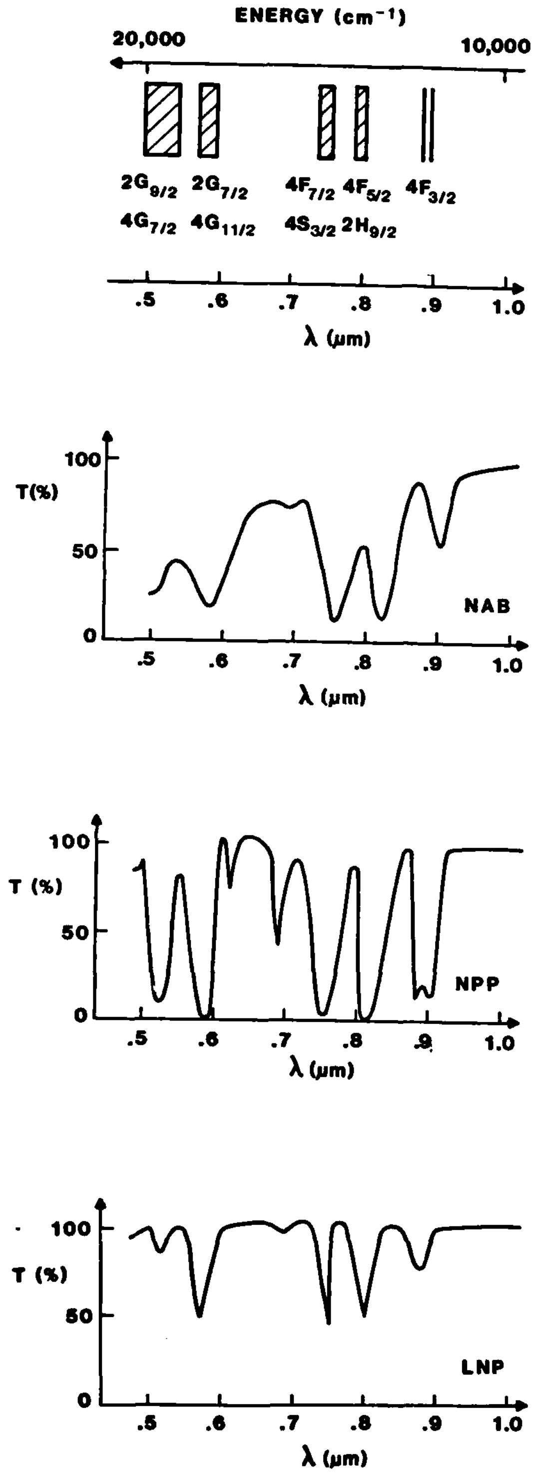
3.1 Initial Optical Characterization of Crystals Grown

Transmission spectra of the samples of the three types of crystals mentioned above were measured by using a Beckman spectrophotometer (Model DK-2A) as described previously [Ref. 14]. Typical examples are given in Figure 6 together with an approximate energy-level diagram of the Nd^{3+} ion for labeling the absorption bands. These transmission data compare well with published results [Ref. 15] in the literature. In the case of NAB, however, there is a broad band absorption near the short-wavelength region of the visible spectrum. This behavior is thought to be either due to lead in the crystal because of the growth procedure using lead fluxes, or due to increased density of scattering centers (less perfect crystal appearance).

The fluorescence measurements on the samples were performed by the experimental setup described previously [Ref. 14]. Typical results for the 1.05 m emission is shown in Figure 7. The lifetimes defined at e^{-1} points of the fluorescence decay curves are also given in Figure 7. The corresponding values taken from published literature [Ref. 5] are given in parenthesis for comparison purposes. When other lanthanides are added in the crystal to replace some of the Nd^{3+} ions, fluoresence lifetime increases as seen in Figure 8. The dependence of fluoresence lifetime on the Nd^{3+} has been analyzed previously [see, for example, Ref. 16].

3.2 Experimental Laser Setup and Flash-Lamp Power Supply

An experimental laser system was built using miniature optical components. These components were partly obtained from Ealing Corp. and partly machined in-house. The laser setup is seen in Figure 9 together with



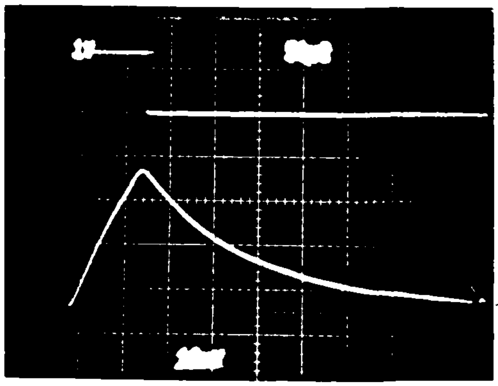
(a) Pump bands

(b) NAB

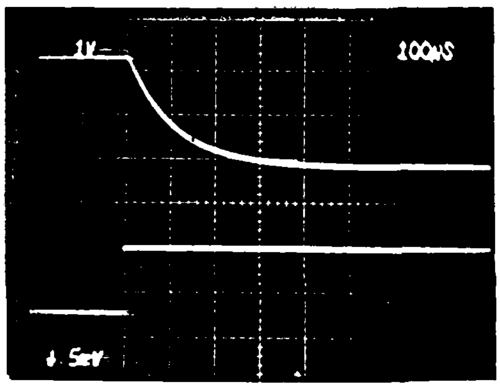
(c) NPP

(d) LNP

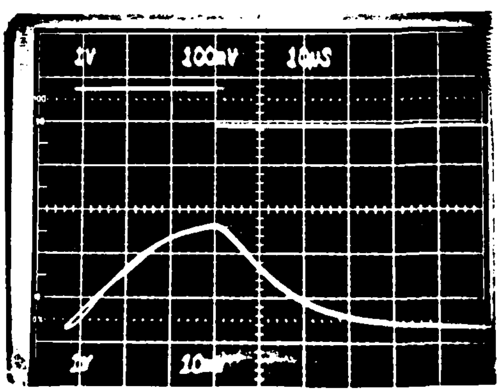
Figure 6. Transmission spectra of NPP, LNP, and NAB crystals. Pump bands of Nd^{3+} are given on top for labeling purposes.



NPP Crystal.
τ = 140 μs (120)

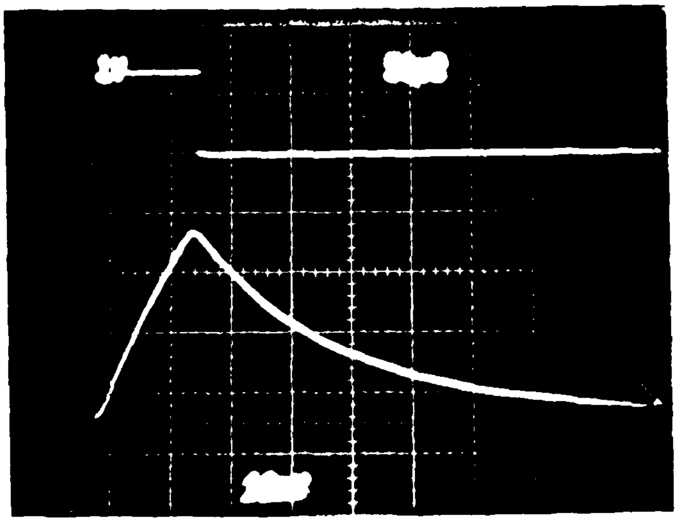


LNP Crystal.
τ = 140 μs (120)

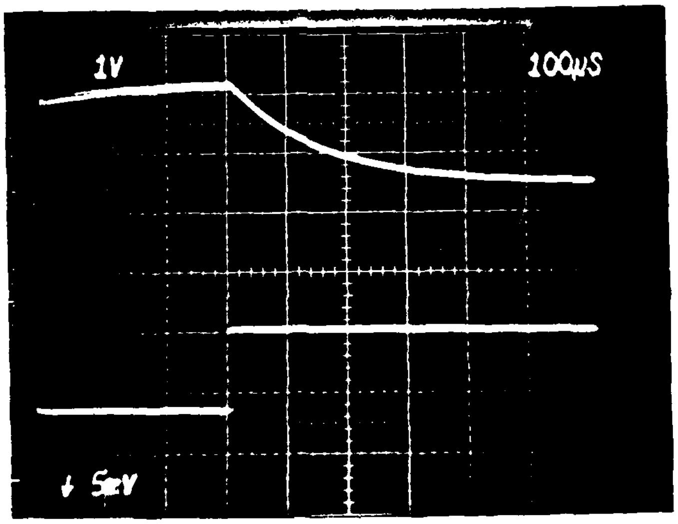


NAB Crystal.
τ = 16 μs (20)

Figure 7. Fluorescence lifetime of Nd^{3+} in crystals of NPP, LNP and NAB grown in-house. Values in parenthesis are publsihed values (Ref. 5) in the literature.

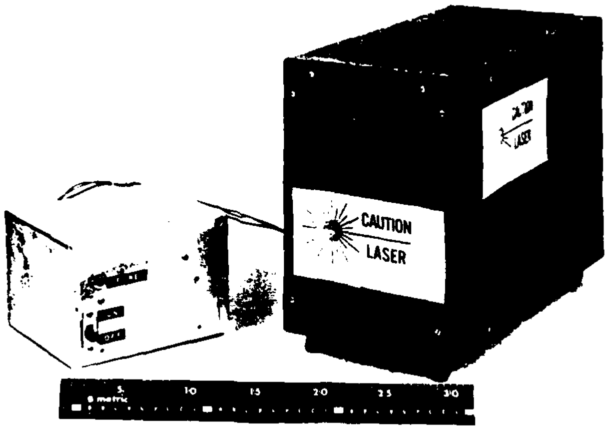


(a) NPP Crystal.

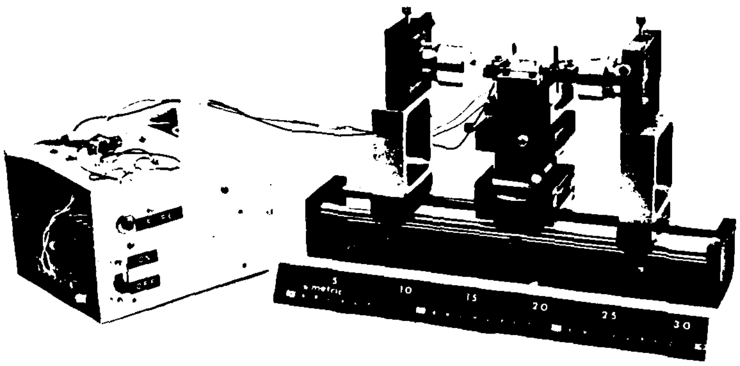


(b) $Nd_xLa_{1-x}P_5O_{14}$ Crystal.

Figure 8. Effect of decreasing Nd^{3+} concentration on fluorescence lifetime for NPP and $Nd_xLa_{1-x}P_5O_{14}$ (x = 0.75).



(a)



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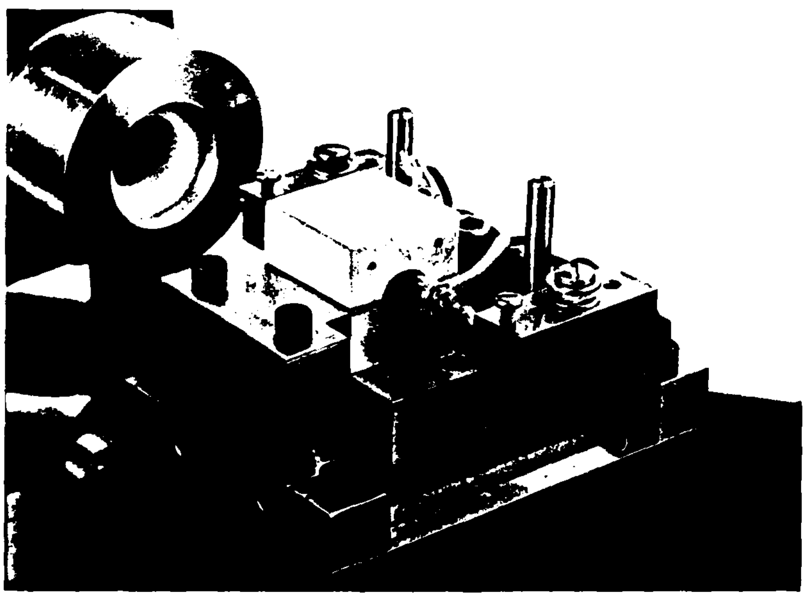


(c)

Figure 9. Experimental laser setup and flash-lamp power supply with and without cover; and laser head showing pump cavity, flash-lamp, and NPP laser rod.

the power supply for the flash lamp. This system is designed to allow the laser rod, pumping cavity, and flash lamp to be moved separately with many degrees of freedom. This enables one to optimize of the laser output.

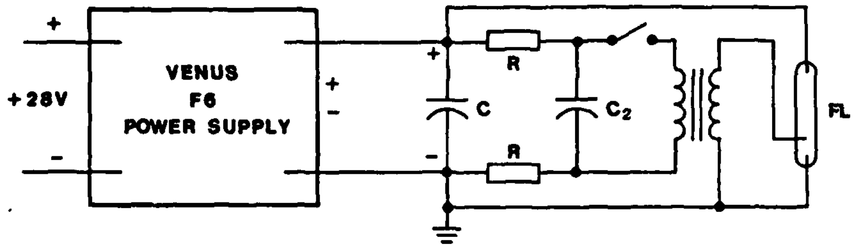
Several power supply circuits were used to discharge the flash lamp; two examples are given in Figure 10. As seen, both series and external triggering were used. External triggering has been found to be more advantageous because it gives better pulse shapes (shorter and with less oscillations), as seen in Figure 11; for doing this, a smaller triggering transformer is needed. However, a disadvantage to external triggering is that it requires a high-voltage terminal around the flash lamp. One of these power supplies with external triggering is shown in Figure 9.

The flash lamps used for the tests were obtained from EG&G, Xenon Corp., and Mauser Electronics (supplier). All of the lamps were either too large for our application or had poor performance. In general, good quality experimental miniature flash lamps seem to be in short supply.

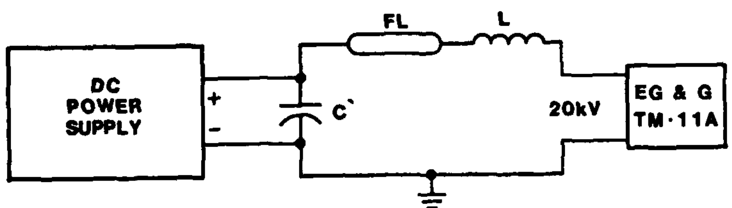
3.3 Initial Results on the NPP Laser Output

For initial characterization of the lasing behavior of Nd^{3+} materials, a 2 x 2 x 20 mm NPP crystal was used in the experimental laser system described above. During this initial study, the laser output energy per pulse, transverse mode structure of beam, and temporal behavior of output pulse were analysed.

The highest output energy obtained from the system was 48 mJ per pulse for 16 J of electrical input energy. For this measurement of output energy, the cavity length was 8 cm, and the mirror reflectivities and curvatures were R_1 = 100%, R_2 = 65%, and r_1 = 10 cm, r_2 = 1 meter, respectively. The transverse mode structure was highly multimode, and the far-field beam angle was about 1°. To improve the transverse mode behavior, a flat output mirror was used with 85% reflectance. This new mirror resulted in an output energy of 35 mJ per pulse with the same 16 J of input electrical energy. The transverse mode structure in this case was improved, as seen from Figure 12a which gives the mode structure at 0.5 meter from the output mirror. The near-field mode structure of a similar beam at the output mirror is seen in Figure 12b which also shows the effect of inhomo-

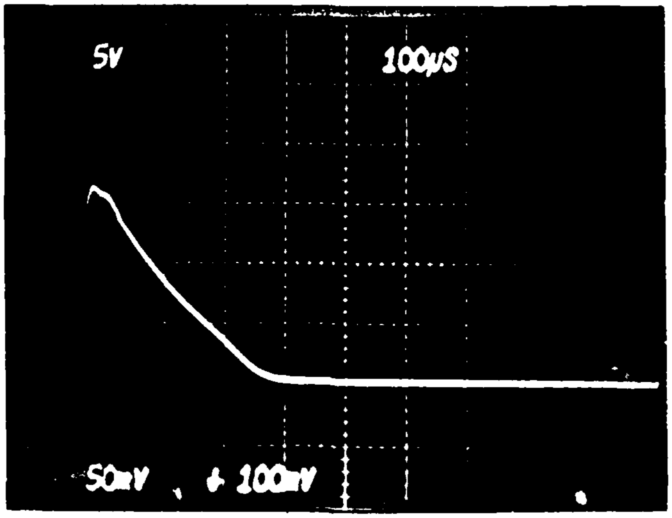


(a) External triggering.

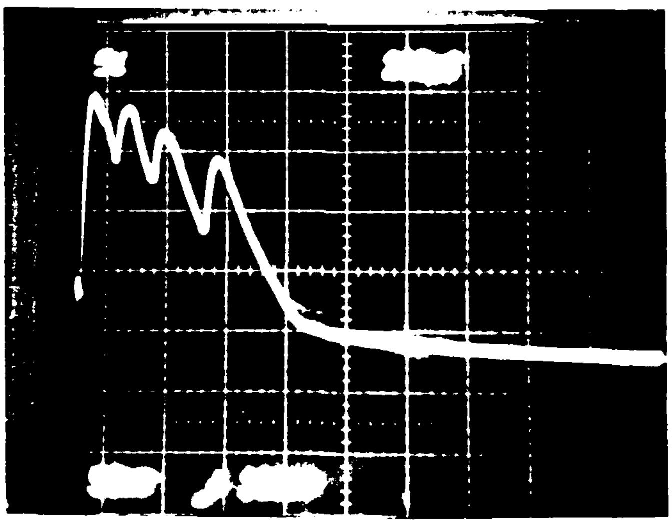


(b) Series triggering.

Figure 10. Schematic of flash-lamp light discharge circuits with external and series triggering.

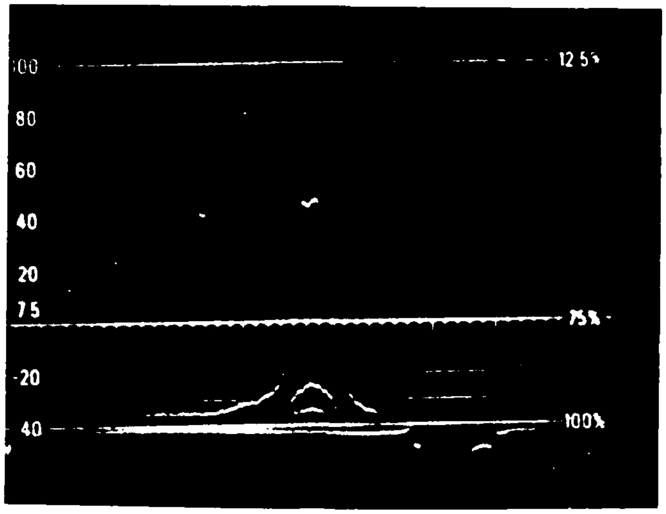


(a) External triggering.

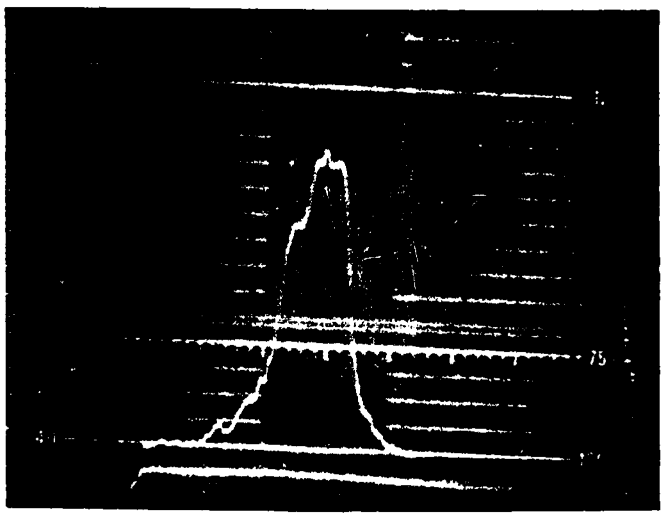


(b) Series triggering.

Figure 11. Light output pulses of flash lamp with external and series triggering.



(a) At 0.5 meter from output mirror.

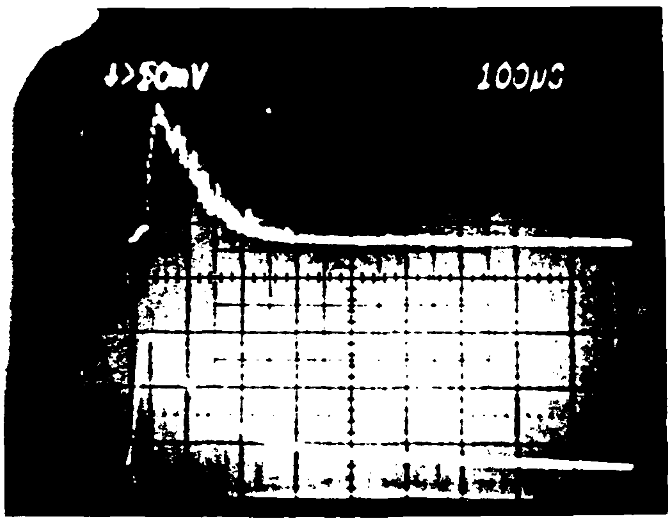


(b) At output mirror (without camera lens).

Figure 12. Transverse mode structure of a 2 x 2 x 10 mm NPP crystal in laser setup shown in Fig. 9 was used with $R_1 = 85\%$, $r_1 = 100$ cm, at 0.5 meter away from output mirror and at output mirror.

geneous pumping of the single elliptical cavity using only one flash lamp. The setup used to obtain the mode structure contained a Newvicon* camera and waveform monitor.

The temporal behavior of the laser output pulse was studied by a setup similar to that used for measuring fluorescence lifetime. The pulse length at FWHM was measured to be about 100 μs, and the pulse shape was the same as that of the flash lamp output as seen from Figure 13. To shorten the laser pulse length, the flash-lamp discharge circuitry will be improved to give shorter pump pulses.



(a) Laser.

(b) Flash-lamp.

Figure 13. Temporal behavior of laser output pulse and flash-lamp output pulse.

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* Koyo CCTV Camera, TVC-5210-25 with $Zn_xCd_{1-x}Te$ target.

4. PLANS FOR NEXT QUARTER

a. Obtain vitreous carbon crucibles of proper shape for growth of NPP crystals and produce large crystals of good optical quality for fabrication of laser rods.

b. Produce crystals of LNP by top-seeded growth techniques.

c. Study fluorescence properties of crystals under different environmental and excitation conditions.

d. Study emission spectra of Nd^{3+} in the crystals under different conditions, using newly purchased photomultiplier tube (Type S-1).

e. Using comparisons of absorption and emission spectra measurements, evaluate magnitude of emission cross sections of Nd^{3+} in crystals.

f. Design a miniature laser system with minimum moving components.

g. Improve output pulse shape and efficiency of flash-lamp discharge circuitry.

h. Make an optimization study (both modeling and experimental) of laser output behavior.

i. Study effects of crystal heating on laser output.

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